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14. ABSTRACT

This report results from a contract tasking University of Strathclyde as follows:

The contractor shall characterize the dielectric signature of the hydrated lightly coated oxide surface, and investigate the effect of various solvent systems on bond durability. The contractor shall carry out ageing studies using hydraulic fluid, aviation fluid, de-icing fluid and urea solutions to explore the way in which ageing is influenced by these media. The outcome of this study shall be a definition of the rate and potential mechanisms of attack of these common fluids.

The contractor shall characterize the ageing of boron fiber/epoxy aluminum bonded structures taking into account that the electrical conductivity of the boron fibers is intermediate between those of carbon and glass. As a result the approach to measurements used with either the carbon fibers or the aluminum structures is not appropriate with these structures. An electrode structure has been constructed which allows investigation of the dielectric properties of these structures, but the effects of moisture and ageing in high humidity environments has not been evaluated. Structures have been created and it is planned to carry out ageing studies over the next two years. Boron fiber composites are used in repair of damage on the skins of aluminum fabricated aircraft. This study will demonstrate the usefulness of the method for the characterization of ageing in these bonded structures.

15. SUBJECT TERMS
EOARD, Aging Aircraft, Composites, Non-destructive inspection

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Non-destructive evaluation of adhesive bonded structures using dielectric methods

A Final report on contract F61775-00WE060

for

European Office of Aerospace Research and Development

by

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Declarations

1. The contractor, University of Strathclyde, hereby declares that, to the best of its knowledge and belief, the technical data delivered herewith under Contract No. F61775-00-WE060 is complete, accurate, and complies with all requirements of the contract.

DATE: 14/10/2002

Name: [illegible]

Title of Authorised Official: Head of Department

2. I certify that there were no subject inventions to declare as defined in FAR 52.227-13, during the performance of this contract

DATE: 14/10/2002.

Name: [illegible]

Title of Authorised Official: Head of Department

FINAL REPORT

Work on the project titled Non-destructive Evaluation of Adhesively Bonded Structures Using Dielectric Methods in the past year has led to the conclusion of the study into the effects of various aviation fluids on the dielectric and mechanical properties of adhesively bonded aluminium joints. Ageing of the aluminium joints has been carried out at 65°C in a variety of solvents including hydraulic fluid, aviation fuel, propylene glycol (de-icing fluid) and dichloromethane (paint stripper), held at room temperature, and water based solvents; de-ionised water, simulated sea-water and a urea/water solution. The adhesive used to bond the joints was AF-163, a modified epoxy, which is widely implemented for load bearing aerospace joints, requiring high strength and excellent environmental durability. This particular study has been ongoing for two years and has shown some very distinct behaviour patterns.

From this study we have found that solvents of a highly polar nature have the most pronounced effect on the dielectric behaviour of adhesive joints. In particular water based solvents (dielectric permittivity of 80) result in an increase in the dielectric permittivity and loss measured throughout the ageing period. Ingress of water into the bonded joints has been monitored and a correlation with the loss in adhesive strength as a result of moisture ingress has been produced. From the correlation curves we can say that as water enters the bonded joints there is a definite loss in both shear strength and interlaminar fracture toughness of the adhesive, with the latter displaying a more pronounced effect. Joints aged in de-ionised water and urea solution show very similar dielectric and mechanical behaviour indicating the effects observed are due solely to the water, with the urea having virtually no effect on the overall behaviour of the joints. In contrast however joints aged in simulated sea water have shown quite dramatic behaviour. Excessive corrosion of the aluminium substrates has been observed on these joints. Salt deposition within the bondline of the joints has led to a widening of the bond, leading to increased ingress of water which has ultimately led to hydration of the interfacial oxide layer. The hydrated product formed does not promote good adhesive properties and so leads to severe degradation of the bonded joints by displacing the adhesive from the oxide interface, in addition to causing severe corrosion on the inside of

the aluminium substrates. This effect has led to several of the joints used in this study simply falling apart before additional mechanical testing could be carried out.

Ageing of joints in dichloromethane has proved to have the most detrimental effect on the mechanical properties of aluminium (and carbon fibre) bonded joints. Ingress of dichloromethane into bonded joints results in the solvent penetrating the polymeric adhesive, swelling the 3D network then leaching of unreacted products occurs followed by degradation of the adhesive by chemical scission of the network. In this case the aluminium joints had lost roughly 75 percent of the mechanical strength after only 50 days ageing at ambient temperatures. Ageing was stopped after 150 days when all mechanical strength was lost. The decrease in mechanical strength of these joints was accompanied by decrease in the dielectric permittivity of the joints, and again correlation curves were produced to show the relationship between dielectric permittivity and mechanical strength.

Solvents that do not exhibit such high dielectric constants do not effect the dielectric permittivity and loss to such an extent as water or dichloromethane. In particular ageing of bonded joints in solvents like aviation fuel, hydraulic fluid and propylene glycol has been shown to have so significant effects on the dielectric permittivity of the joints. As a result, these joints were able to retain a significant amount of their mechanical strength in the shear mode, however, in term of interlaminar fracture toughness there was a more pronounced reduction in strength. This might be due to the Teflon inserts (used to facilitate crack initiation) which provide an easier route for solvent penetration within the joint. It should be noted that no corrosion or widening of the bondline was observed for the joints aged in these solvents.

In addition to the study discussed above work has concentrated on the manufacture of a series of carbon fibre composite joints using the AF-163 structural epoxy resin. These joints have been aged at an elevated temperature of 75°C in de-ionised water and systematically spiked to cryogenic temperatures in an attempt to simulate the environment bonded joints experience during flight. Results to date indicate an increase

in water uptake of a Fickian nature, and as with the aluminium joints this increase in water content is accompanied be a decrease in mechanical shear strength. Exposure to cryogenic temperatures does not appear to have any significant effect on the bonded structures at this time, however, further monitoring of this is required.

A second series of aluminium bonded joints was manufactured using Epibond 1590 adhesive which is a two component epoxy paste adhesive, supplied by Vantico. These joints were aged at a temperature of 50°C in de-ionised water in an attempt to characterise the dielectric behaviour. As ageing progresses the ingress of water into the bonded joint can be monitored using dielectric spectroscopy and as before the increase in the permittivity, associated with water both bound to the adhesive and free within the bondline, is accompanied by a decrease in mechanical shear strength of the aluminium joints. At this point in the ageing program there is no sign of corrosion of the aluminium. As well as the bonded joints, the neat adhesive was cured to provided water uptake and mechanical data on the bulk adhesive. The adhesive was also aged in de-ionised water at 50°C and again displayed water uptake profiles that were Fickian in nature. Again work is still ongoing in this particular study.

In conclusion the work carried out so far in this area has shown that dielectric spectroscopy can be used to monitor the effects of water uptake and degradation within adhesive bonded structures and has potential to be a non-destructive evaluation technique.